# OXYGEN RELATIONSHIPS IN INTERMITTENT SAND FILTRATION OF WASTEWATERS

Thesis by

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### ABSTRACT

A model for some of the many physical-chemical and biological processes in intermittent sand filtration of wastewaters is described and an expression for oxygen transfer is formulated.

The model assumes that aerobic bacterial activity within the sand or soil matrix is limited, mostly by oxygen deficiency, while the surface is ponded with wastewater. Atmospheric oxygen reenters into the soil after infiltration ends. Aerobic activity is resumed, but the extent of penetration of oxygen is limited and some depths may be always anaerobic. These assumptions lead to the conclusion that the percolate shows large variations with respect to the concentration of certain contaminants, with some portions showing little change in a specific contaminant. Analyses of soil moisture in field studies and of effluent from laboratory sand columns substantiated the model.

The oxygen content of the system at sufficiently long times after addition of wastes can be described by a quasi steady-state diffusion equation including a term for an oxygen sink. Measurements of oxygen content during laboratory and field studies show that the oxygen profile changes only slightly up to two days after the quasi-steady state is attained.

Results of these hypotheses and experimental verification can be applied in the operation of existing facilities and in the interpretation of data from pilot plant-studies.

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# CHAPTER 1 INTRODUCTION

#### 1-1 Background

Intermittent sand filtration is a method of wastewater treatment in which the liquid is applied intermittently to a natural or artificial bed of porous media and allowed to percolate through it to underdrains or to the ground-water table. Its use for the disposal of wastewaters is by no means new. Sedgwick (1) and Kinnicutt, Winslow, and Pratt (2) refer to laboratory studies by Sir Edward Frankland published in 1870 by the Rivers Pollution Commission of Great Britain. These studies, the first on intermittent percolation, showed that the process produced an effluent that was "remarkably purified."

Several important conclusions were drawn from Frankland's studies. He observed that the word "filtration" was really a misnomer since not only physical but also biological processes are involved. The experimenters recognized that aerobic bacterial action was needed to oxidize wastes and to minimize clogging. Hence sewage, which is usually low in dissolved oxygen, had to be applied intermittently to allow atmospheric oxygen to reach the microoganisms.

The first practical application of intermittent sand filtration was by J. Bailey-Denton who constructed four

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beds totalling 20 acres at Merthyd Tydvil, Wales in 1871. Raw sewage was applied to each bed for six hours daily at a net rate of 60,000 gallons per acre per day (0.18 ft per day). The effluent was collected by an underdrain system. Application was later reduced to 16,000 gallons per acre per day by addition of more land. The original plant worked well, however, and it was a practical demonstration of Frankland's process (2).

The first studies in the United States on intermittent sand filtration were conducted at the Lawrence (Massachusetts) Experiment Station, beginning in 1887. Results indicated again the need for intermittent application of sewage and showed a significant reduction in bacterial numbers. As a result of these studies, the towns of Framingham and Brockton soon built large municipal filters and other cities followed suit(1).

The many studies on intermittent sand filtration since Frankland's original experiments have well established the need for intermittent application of wastes to allow sufficient atmospheric aeration and to avoid clogging. Spreading basins operated continuously become anaerobic because any dissolved oxygen that may be present in wastewater is insufficient to satisfy the biochemical oxygen demand.

Although the importance of sufficient aeration during waste degradation by intermittent percolation has

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been accepted by sanitary engineers, the phenomena associated with reoxygenation remain obscure. With the increased interest in tertiary wastewater treatment and in the recharge of ground-water basins for water reclamation, the mechanisms and rates by which oxygen is utilized during percolation and resupplied from the surface deserve attention.

Adequate descriptions of the physical processes are equally lacking. Most published studies approach intermittent sand filtration as a "black box," recording only changes in effluent quality caused by variations in influent type, loading and strength, and frequency of application. There have been few attempts to interrelate physical and biological processes, but such a description is necessary to understand transfer and bacterial utilization of oxygen.

It was the aim of this research, therefore,

- To investigate deoxygenation and reaeration in soil systems used for stabilization of wastewaters, and
- To describe other processes in intermittent sand filtration as a prelude for understanding oxygen transfer and bacterial utilization.

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# 1-2 Use of Intermittent Sand Filtration

Early intermittent sand filters treated either raw or settled sewage or septic-tank effluent. Since these filters required large land areas (e.g. about five acres per 1000 people) they were largely replaced, as populations increased, by trickling filters or other processes requiring less land. In recent years, the need for groundwater replenishment and for advanced waste treatment has rekindled interest in intermittent sand filtration. When used for these purposes, intermittent sand filtration follows secondary treatment. Hence, the future of this process appears to lie in tertiary, rather than primary treatment.

Intermittent sand filtration following secondary treatment is being used successfully for ground-water recharge in several locations in the United States and other countries. Noteworthy examples are installations at Whittier Narrows, California and Tel Aviv, Israel.

The basins at Whittier Narrows are part of a demonstration project for reclaiming domestic wastewaters by activated-sludge treatment followed by surface spreading for ground-water recharge. Approximately 15 million gallons per day (mgd) of domestic wastes are presently being treated. The Israel project is designed to reclaim wastewater from the Tel Aviv metropolitan area. Wastes are treated in stabilization lagoons before ground-water recharge through dune sands (3). The facility is being planned for the year 1990 when the design population will exceed one million people and expected flows will be over 100 million cubic meters per year (72 mgd).

Advanced waste-treatment methods are needed to remove nutrients such as nitrogen and phosphorus from wastewaters in order to avoid algal blooms in receiving streams. Intermittent sand filtration may be used as the first step in a nitrification-denitrification scheme for removing nitrogen from wastewaters because it encourages the growth of nitrifying bacteria. Because intermittent sand filtration does not remove phosphorus, some other operation will be needed for phosphorus removal. Levin (4) has described modifications to the activated-sludge process for phosphorus removal.

#### 1-3 Clogging

Since infiltration rates tend to decrease as a result of clogging, consideration of the causes of clogging is required to understand the necessity for intermittent application. Allison (5) showed that clogging was not due to purely physical causes such as soil-aggregate breakdown but was caused by microorganisms. Gupta and Swartzendruber (6) confirmed Allison's work when they found that hydraulic

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conductivity for boiled deionized water decreased markedly during flow and that no decrease occurred when a 0.1 percent phenol solution was used. In addition, no clogging took place when bacteria at the soil surface were less than 400,000 per gram of soil, but that drastic reduction occurred above this concentration. Since the volume of bacteria constitutes only a small portion of the pore space, Gupta and Swartzendruber (6) concluded that associated products rather than the bacteria themselves cause clogging.

McGauhey and Winneberger (7), studying septic tank percolation systems, claimed that sulfide formation in soil systems caused clogging through precipitation of ferrous sulfide. Nevo, with Avnimelech (8) and with Mitchell (9), passed NH<sub>4</sub>NO<sub>3</sub> in tap water through sands to which organics had been added. They disputed McGauhey and Winneberger's claim and believed that sulfide formation was only an indicator of anaerobic conditions. Mitchell and Nevo (9) stated that polysaccharide accumulation was highly correlated to clogging, but Avnimelech and Nevo (8) concluded that polyuronides were responsible.

Thomas, Schwartz, and Bendixen (10) found that phosphate and total organic matter (chemical oxygen demand) accumulated in soil during clogging, in addition

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to iron, sulfide, polysaccharide, and polyuronide. Of these parameters, only organic matter declined as the infiltration rate was partially recovered during a rest cycle.

The substances causing clogging are most readily degraded aerobically by fungi and bacteria. Because the oxygen demand of wastewaters is many times the dissolved oxygen (if any) in the wastewater, oxygen must be supplied to the soil system. The most practical way to do so is to allow air to enter the system during a rest cycle.

Amramy (3) notes that while many of the substances causing clogging are produced anaerobically, polysaccharides cannot be produced without oxygen. Since polysaccharides accumulate even during aerobic conditions, intermittent operation of percolating beds serves still another function, namely, to force the bacteria to respire endogenously and thereby to utilize and stabilize some of their own waste products.

### 1-4 Spreading Basin Operation

In practice, effluent from a primary or secondary wastewater treatment plant is spread on a basin and the basin is allowed to drain until no liquid is ponded on the surface. The basin is then "rested" before further application of treated wastes to allow oxygen from the atmosphere to diffuse into the soil.

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The cycle time (ponded plus resting) for a given installation has been determined in the past by trial and error from observations of clogging rates and effluent quality. These studies have resulted in a variety of loading methods.

Amramy (3), applying effluent from a series of lagoons on a dune sand (effective size 1.15 mm), could maintain an approximately constant infiltration rate by using a cycle of ten days, five wet and five resting. For safety, he suggested five or six wet days and twice as many resting days.

Robeck <u>et al</u> (11) noticed that effluent quality was improved by reducing the total cycle time to between four and eight hours.

Others, e.g. Laverty, Stone, and Meyerson (12) and McMichael and McKee (13) used 24-hour cycles with success. McMichael and McKee maintained the infiltration rate in test basins by adjusting the volume spread once each day so that the wet time did not exceed 12 hours.

These examples indicate that installations have successfully used cycles of between four hours and about two weeks. The results of this research should provide some insight into the required cycle times.

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### CHAPTER 2

# PROCESSES IN INTERMITTENT SAND FILTRATION 2-1 Redistribution of Soil Water

If Darcy's law is assumed valid for unsaturated flow of an incompressible liquid in porous media, continuity may be written for vertical flow as

 $\frac{\partial \Theta}{\partial t} = \frac{\partial}{\partial z} \left( \kappa \frac{\partial p}{\partial z} \right) - \frac{\partial \kappa}{\partial z}$ (2-1)

where

 $\Theta$  = soil water content,  $L^3/L^{3^*}$ 

t = time

- z = distance measured positively downward, L
- K = capillary conductivity, L/t
- p = soil water pressure expressed as height of water, L

Equation 2-1 is difficult to solve because both K and p (sometimes called capillary head) depend on moisture content. The problem is compounded because the relation between  $\Theta$  and p is not single-valued but depends on the history of the media (14). Equation 2-1 has been solved

\* Dimensions are given in terms of mass (M), length (L), and time (t).

only for the simplest cases. Even then, solutions have required simplifying assumptions or the use of digital computers.

Some work has been done on the problem of water infiltrating a soil (15, 16) and on drainage of initially saturated porous materials (17, 18, 19), but the literature on redistribution of soil water after infiltration is scarce indeed. Biswas, Nielsen, and Biggar (14) have observed that the distribution pattern depends on soil type and depth of water added (Fig. 2-1). For coarse materials or large additions of water, the medium was saturated to a short depth as infiltration ended. Further redistribution proceeded with an immediate moisture loss near the surface and a reduction in the zone of saturation (Fig. 2-la). For fine-textured soils whose retained , moisture varied gradually with increasing height above a water table or for shallow depths of water, moisture contents never approached saturation, except at the soil surface (Fig. 2-1b). For fine-textured soils, infiltration (the passage of water into the soil across the interface of water and soil, or air and soil) is often the limiting or controlling mechanism rather than percolation (the vertical downward movement of water within the soil).

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Fig. 2-1 Hypothetical soil-water profiles during redistribution of water. In a, the sand is coarse; in b, the sand is fine. At time  $t_0$ , for profile a, the soil is saturated from the surface to the lower edge of the crosshatched area. At time  $t_2$ , the zone of constant water has diminished to that represented by the cross-hatched area. After Biswas, Nielsen, and Biggar (14). -12-

## 2-2 Mechanisms for Removal of Pollutants

Processes acting on pollutants during intermittent sand filtration may be divided into two categories, viz. physical-chemical processes that remove pollutant from the percolate and biological processes that stabilize the wastes. These two categories are reviewed briefly in the following sections.

### 2-2-1 Removal of Wastes from Percolating Liquid

In the early days of intermittent sand filtration when raw sewage or settled sewage was applied to sand beds, straining was an important removal mechanism and the suspended matter thus retained tended to form a mat on the filter bed. This build-up of organic matter impeded infiltration and hindered oxygen transfer into the soil. Now, when intermittent sand filtration is used it generally follows secondary treatment and constitutes advanced waste treatment or initiates wastewater reclamation processes for replenishment of the ground water. Suspended-solids content of the typical water presently used for wastewater reclamation is substantially below that of raw sewage or primary effluent. Hence, suspended-matter removal by intermittent sand filtration is less important now than in former years.

While the soil is draining, some polluting material is removed from the percolating liquid by physical adsorption on soil surfaces, some by diffusion into stagnant zones, and some by biological assimilation and biosynthesis. Concurrently, other substances are being desorbed from soil surfaces, removed from stagnant zones, and excreted as a result of biological metabolism. In intermittent sand filtration the substances being adsorbed or otherwise removed from percolating water may be dissolved, colloidal, or suspended solids. In general, they are complex organic compounds or ammonium ions. The substances being returned to the percolating water are generally dissolved minerals or stabilized organics.

The extent of biological utilization of organic substances during percolation depends both on the nature of the component being considered and on oxygen availability. Substances that are readily biodegradable will be almost completely stabilized while oxygen is still present. More resistant compounds may be adsorbed by biological films and metabolized slowly. Some compounds may be essentially unaffected by percolation through biologically rich porous media.

### 2-2-2 Biological Processes

The object of intermittent sand filtration, in addition to maintaining high rates of hydraulic acceptance, is to stabilize wastes to innocuous end products.

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Under aerobic conditions, the major end products in biochemical stabilization of carbonaceous and nitrogenous substances are water, carbon dioxide, bicarbonates, sulfates, phosphates and nitrates. In the absence of oxygen, carbonaceous material may be converted to carbon dioxide and methane, but nitrogenous substances degrade only to ammonia, and cannot be oxidized to nitrate.

Ammonia is nitrified in two steps by autotrophic bacteria which do not use organic carbon. <u>Nitrosomonas</u> first mediate the oxidation of ammonia to nitrite and <u>Nitrobacter</u> complete the process by catalyzing the oxidation of nitrite to nitrate. Thimann (20) considers nitrite formation as a pair of linked reactions, the second of which has two alternatives:

 $2 \text{ NH}_4^+ + 4 \text{H}_2 0 \longrightarrow 2 \text{NO}_2^- + 4 \text{H}^+ + 12 (\text{H}) (2-2)$ 

12 (H) + 3  $O_2 \longrightarrow 6H_2O$  (2-3a)

12 (H) + 3 
$$CO_2 \longrightarrow 3(CH_2O) + 3H_2O$$
 (2-3b)

The proportion of reaction 2-3a to 2-3b can be estimated from studies that measured the ratio  $NH_4^+$  oxidized:  $CO_2$ reduced. Thimann (20) has obtained values ranging from 20 to 100 for this ratio and cites values from 15 to 53. A ratio of 15 corresponds to 21.5 of reaction 2-3a to one of reaction 2-3b; a ratio of 100 corresponds to 149 of reaction 2-3a to one of reaction 2-3b.

The reactions for nitrate formation can be written as:

$$2NO_{2}^{-} + 2H_{2}O \longrightarrow 2NO_{3}^{-} + 4(H) \qquad (2-4)$$

$$4(H) + O_{2} \longrightarrow 2H_{2}O \qquad (2-5a)$$

$$4(H) + CO_{2} \longrightarrow (CH_{2}O) + H_{2}O \qquad (2-5b)$$

Schön (21) has measured the ratio of  $NO_2^-$  oxidized:  $CO_2^-$  reduced. His value corresponds to 36 of 2-5a to one of 2-5b.

In aerobic activity involving heterotrophic bacteria, oxygen is the terminal electron acceptor of the electrontransfer chain. In anaerobiosis, other elements are the acceptors and are reduced. These elements include nitrogen in nitrate and nitrite, sulfur in sulfate, and carbon in organic compounds.

Nitrate reduction (denitrification) can be either assimilatory or dissimilatory. In assimilatory denitrification, nitrogen is reduced to minus-three valence for incorporation in the cell structure. Dissimilatory nitrate reduction produces  $N_2$ ,  $N_2O$ , and NO. The formation of the latter three gases would result in a reduction of total nitrogen in the soil water. The studies on wastewater reclamation by McMichael and McKee (13) did not show a nitrogen loss although denitrification is known to occur in soils (22).

The decomposition of organic materials with sulfate as the terminal electron acceptor produces sulfide or elemental sulfur. Sulfide is formed in soil systems only after clogging has begun (10). The test basins used by McMichael and McKee (13) have not clogged although they have been in operation for more than four years. Hence it would seem that significant sulfate reduction can be eliminated in at least some intermittent filters.

Although carbon as an electron acceptor (fermentation) results in reaction rates that are considerably slower than found under aerobic conditions (23), fermentation may account for some stabilization in an intermittent sand filter because only the top few feet are aerobic and even this area becomes anaerobic after the surface is ponded.

The presence or absence of significant anaerobic bacterial activity at depths permanently devoid of oxygen should be considered when one is studying oxygen transfer during intermittent sand filtration. This matter is important because the movement of gases produced at anaerobic depths (nitrogen, methane) will influence the movement of oxygen gas into the aerobic zone. The effect of other gas movements on the movement of oxygen will be discussed below.

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From measurements showing that more organic nitrogen was collected in a laboratory-filter effluent than was added, Morgan and Gilcreas (24) have suggested that nitrogen fixation occurs in the upper portion of sand filters. 2-3 A Model for the Processes in Intermittent Sand Filtration

When secondary treatment plant effluent infiltrates into a soil, substances are removed from the percolating liquid as described previously when the incoming liquid displaces whatever liquid existed previously in the soil. Although the substances in the incoming liquid are affected by bacterial activity, stabilization of some components is limited because the liquid percolates through the soil too quickly for the microoganisms to assimilate and completely degrade the wastes. In addition, bacterial activity is limited by availability of oxygen.

Therefore, immediately after ponded water has completely infiltrated into the soil, the composition of the soil water with respect to those components not assimilated or adsorbed is essentially constant for some depth into the soil. During subsequent drainage, air enters into the soil and aerobic activity increases.

Secondary effluent is complex but can be characterized by analyses for the carbonaceous and nitrogenous components (carbohydrates, fatty acids, protein). At most wastewater treatment plants, almost all of the

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nitrogen is present as ammonia or organic matter; at the Whittier Narrows Water Reclamation Plant (activated-sludge plant) the effluent, which is to be used for ground-water recharge, contains 10 to 15 percent of the total nitrogen as nitrate.

The extent of aerobic activity can be related to the increase in nitrate in the soil moisture. Other chemical determinations could be used for this purpose, but nitrate determination is convenient and nitrification is an important function. Because of limited stabilization while the surface is ponded, nitrate content of soil water can be expected to be essentially constant throughout the soil profile at the end of infiltration and to increase as bacterial activity stabilizes the influent. The first increase in nitrification will occur near the . surface because atmospheric oxygen is first available at the surface. Nitrification will progress toward greater depths as oxygen becomes available to nitrifying bacteria. However, there is a depth below which there is no oxygen. Incoming wastewater that passes into depths permanently devoid of oxygen does not become further nitrified.

Fig. 2-2 shows hypothesized changes in nitrate with time and depth in an intermittent filter. At  $t_0$  infiltration has just been completed and the nitrate content of the soil water is roughly constant with depth, but some-

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Hypothesized variation of nitrate in soil water Fig. 2-2

what higher than the nitrate content of the secondary effluent applied because of the nitrification that occurred during drainage. The sketches for times  $t_1$  to  $t_3$  show the change in nitrates with time. Time  $t_3$  is just before more wastewater is spread on the filter surface.

The incoming waste displaces the pellicular water and dispersion changes the shape of the nitrate wave, as shown in sketches for  $t_A$  to  $t_7$ .

A plot of the variation of nitrate content at depth "d" of Fig. 2-2 is shown in Fig. 2-3. Here it is clear that the nitrate content varies with time.

#### 2-4 Oxygen Transport

The equation of continuity for gas A in a porous medium can be written as

$$\frac{\partial \in c_A}{\partial t} + \frac{\partial N_{AZ}}{\partial z} = R_A \tag{2-6}$$

where  $\epsilon$  = gas porosity, dimensionless

 $c_A$  = molar concentration of gas A, moles/L<sup>3</sup>  $\neq$  = time, t

 $N_{Az}$  = molar flux of gas A in z direction with respect to stationary coordinates, moles/L<sup>2</sup>t

z = distance, L

 $R_A$  = molar rate of production of species A, moles/tL<sup>3</sup>





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The molar flux of A can be expressed as the sum of two fluxes, one resulting from bulk flow, the other from diffusion (25). For a three-component system the molar flux of component A is

$$N_{AZ} = X_{A} \left( N_{AZ} + N_{BZ} + N_{CZ} \right) - c \mathcal{D} \frac{\partial X_{A}}{\partial Z}$$
(2-7)

in which

 $x_A$  = mole fraction of gas A, dimensionless

- $N_{B_2}$  = molar flux of gas B in z direction, moles/L<sup>2</sup>t
- $N_{c_{z}} = \text{molar flux of gas C in z direction,}$ moles/L<sup>2</sup>t

 $\mathscr{D}$  = effective diffusivity,  $L^2/t$ 

The effective diffusivity is less than the diffusivity in free space ( $\mathcal{D}_o$  ) because available cross-section area is reduced and true path length increased by the media. While effective diffusivity depends on factors including pore size and shape, many attempts have been made to relate  $\mathcal{D}/\mathcal{D}_o$  to gas porosity only. The resulting empirical or semi-empirical relationships vary with soil type and moisture content. Moisture, aside from serving as a pore filter, can alter pore shape and block some pores. Typically, the relationships are formulated in expressions such as:

$$\mathcal{D}/\mathcal{D}_o = \eta \in^{\mathcal{T}} \tag{2-8}$$

or

$$\mathcal{D}/\mathcal{D}_{o} = a \mathcal{E} + b \tag{2-9}$$

with  $\eta$ ,  $\sigma$ , a, b constants.

Currie (26,27) has fitted the data for several types of porous media with  $\sigma = 4$ , except at lower porosities, where  $\sigma$  drops below 4. For sand fractions between 0.25 and 0.50 mm, Currie's data can be fitted with  $\gamma = 16$  and  $\sigma = 4$  for  $\epsilon$  greater than 0.2. For  $\epsilon$  less than 0.2 the data points fit the expression

$$D/D_{o} = 0.14 e^{-1.19}$$
 (2-10)

Equations 2-6 and 2-7 (with suitable effective diffusivity) can be used to study bacterial utilization of oxygen and oxygen transfer in a soil system. For this case, gas A may be oxygen and gas B carbon dioxide. Component C can comprise the remaining gases, including nitrogen, hydrogen and methane. Because oxygen is withdrawn rather than produced,  $R_A$  is negative.

One requirement for the solution of equation 2-6 for the transient state is a statement of the initial condition (immediately after the ponded water has completely infiltrated into the soil). The initial condition is difficult to obtain because of the difficulty of assessing the effect of water infiltration on redistribution of gases in the porous medium. In addition, porosity and rate of oxygen uptake vary with time and distance in some complicated manner during infiltration.

It therefore seems expedient to abandon (at least for now) attempts at describing in mathematical terms the transient-state concentration in the soil and to concentrate on the steady-state form of equation 2-6:

$$\frac{dN_{AZ}}{dz} = R_A \tag{2-11}$$

True steady state does not occur in a bacterial system unless substrate is continuously added and growth products removed. However, a quasi-steady state, one in which the oxygen concentration depends only on the effective diffusivity and on the bacterial respiration, can exist at some time if temporal changes in these two variables are sufficiently slow.

To solve the quasi steady-state equation for oxygen transport, a model is needed for the variation of bacterial respiration and of effective diffusivity with soil depth.

Another condition that must be specified before the problem can be analyzed is the relationship between

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 $N_{Az}$ ,  $N_{Bz}$ , and  $N_{cz}$ . Under aerobic conditions and at steady state,  $N_{cz}$ , the flux of nitrogen, hydrogen, and methane, must be zero because none of these gases are produced in the system.

In their reaeration studies, soil scientists have essentially assumed that  $N_{Az} = -N_{Bz}$ . This assumption implies that a mole of carbon dioxide is given off for every mole of oxygen used up.

With this assumption, equation 2-7 becomes

$$N_{az} = -c \mathcal{D} \frac{dx_a}{dz} \tag{2-12}$$

and equation 2-11 can be written as

$$\frac{dN_{AZ}}{dz} = -\frac{d}{dz} \left( c \mathcal{D} \frac{dX_A}{dz} \right) = \mathcal{R}_A$$
(2-13)

In most cases, however, carbon dioxide production does not equal oxygen consumption. The respiratory quotient, the ratio of moles of carbon dioxide produced to moles of oxygen consumed, can be calculated from the stoichiometry of the equations for the oxidation of carbonaceous matter. The respiratory quotients are 1.0 for oxidation of carbohydrate, about 0.7 for fatty acids, and about 0.8 for proteins. Respiratory quotients greater than unity can occur, for example during synthesis of fats from carbohydrates, but they seldom exceed 1.0 under normal circumstances (28). Respiratory quotients for
nitrification are small negative numbers because about 1/40 mole of carbon dioxide is consumed for every mole of oxygen consumed (See Section 2-2-2).

If  $N_{Bz} = -q N_{Az}$  where q is the respiratory coefficient, equation 2-7 becomes

$$N_{AZ} = -\frac{1}{(1 - x_A + q x_A)} \quad c \mathcal{D} \quad \frac{d x_A}{d z} \tag{2-14}$$

and equation 2-11 becomes

$$\frac{dN_{Az}}{dz} = -\frac{d}{dz} \left[ \frac{l}{(l-x_A+qx_A)} c \partial \frac{dx_A}{dz} \right] = \mathcal{R}_A \qquad (2-15)$$

Because the respiratory quotient depends on the substrate and on the oxidation accomplished, its value in intermittent sand filtration is not known, but it is most probably between zero and one. To determine whether the respiratory quotient greatly influences oxygen concentrations, equation 2-11 can be solved with respiratory quotient ( q ) equal to zero (equation 2-15) and with respiratory quotient equal to one (equation 2-13). A simple model suitable for this purpose is one with constant diffusivity and constant oxygen usage to some depth (L), beyond which activity is zero (Fig. 2-4).

Appropriate boundary conditions are

at z = 0 (surface)  $x_A = x_{AO}$ at z = L  $dx_A/dz = 0$ 



Fig. 2-4 Diagram of profile of bacterial-respiration rates for example to determine effect of respiratory quotient on oxygen profile. For constant total gas pressure, c, and these boundary conditions, the solution to equation 2-13 is

$$X_{A} = X_{AO} - \frac{R_{A}}{c \mathcal{B}} L^{2} \left[ \frac{1}{2} \left( \frac{z}{L} \right)^{2} - \frac{z}{L} \right]$$
(2-16)

in which  $\begin{array}{c} x \\ A0 \end{array}$  is the mole fraction of A at the surface and L is the distance at which activity becomes zero (29,30).

For the same conditions and respiratory quotient of zero, the solution to equation 2-15 is

$$In\left(\frac{I-\chi_{AO}}{I-\chi_{A}}\right) = -\frac{R_{A}}{c\mathcal{D}} L^{2}\left[\frac{I}{2}\left(\frac{z}{L}\right)^{2} - \frac{z}{L}\right]$$
(2-17)

A plot of equations 2-16 and 2-17 for two values of  $R_A/cD$  differing by two orders of magnitude shows that the maximum differences in oxygen partial pressures are about 0.015 atmosphere (Fig. 2-5). Because respiratory quotients in intermittent sand filtration are not known ', and because experimental error may be considerable, a respiratory quotient equal to one may be used in analyzing data.

Equation 2-13 can be used to determine effective diffusivity after  $x_A$  and  $R_A$  have been determined. For this purpose, equation 2-13 is integrated once with the boundary condition

at z = L  $dx_A/dz = 0$ 

and rearranged to yield

$$\mathcal{D}(z) = \frac{\int_{z}^{L} R_{A}(\xi) d\xi}{c \frac{dx_{A}}{dz}}$$
(2-18)



Fig. 2-5 Partial pressures of oxygen for example to determine effect of respiratory quotient on oxygen profile.

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in which  $\xi$  is a dummy variable and L is the distance at which oxygen concentration is zero. In effect, diffusivity is the flux rate divided by the gradient.

Whenever diffusivity and respiration rates can be estimated with reasonable accuracy, equation 2-13 can be used to calculate the oxygen profile by integrating twice with the additional boundary condition

at z = 0 (surface)  $x_A = x_{A0}$ 

to yield

$$X_{A} = X_{AO} + \frac{1}{c} \int_{0}^{2} \frac{\int_{z}^{L} R_{A}(\xi) d\xi}{\mathcal{D}(\xi)} d\xi \qquad (2-19)$$

Because L (the depth at which  $x_A$  and, therefore,  $R_A$  become zero) is unspecified, trial values of L have to be used in most cases to obtain a solution where both  $dx_A/dz$  and  $x_A$  are zero at L.

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#### CHAPTER 3

# APPARATUS AND ANALYTICAL PROCEDURES

# 3-1 Soil Columns

Four columns were built so that they could be split axially to remove the soil after they had been operated as laboratory intermittent sand filters. For each column, two acrylic-plastic bars with a cross-section of 1.25 x 3.5 inches were joined to make a combined crosssection of 2.5 x 3.5 inches, and a 1.5-inch diameter hole\* was bored axially through the center (Fig. 3-1). Length was sufficient for a 150-cm sand column. The sand was supported by a 200-mesh stainless-steel screen welded to a 0.125-inch length of 1.75-inch 0.D. stainless-steel tubing with 0.125-inch wall thickness. A 2-cm layer of fine gravel (4mm) was placed above the sand to distribute ', the flow and to prevent scouring. A 1.5-inch I.D. acrylic-plastic tube extending above the column allowed for water depth up to 18 inches (Fig. 3-2).

Apparatus for sampling soil atmosphere was similar to that used by Ritchie (31) and Kimball (32). Twenty sampling probes (Fig. 3-1) were placed along each column.

\* Boring was done by Clark and Wheeler Engineering, Inc., Paramount, California



# Legend:

- 0.032-inch x 0.006-inch wall stainless -steel tube
- 2. Modeling clay
- 3. Solder
- Modified 1/8-inch brass Swagelok male-connector body
- 5. Brass collar
- 6. 1/16-inch diameter hole 1/4 inch deep
- 7. Solder
- 8. Septum seal for gas chromatograph
- 9. Swagelok nut

Fig. 3-1 Section of column showing gas-sampling probe.



Fig. 3-2 Setup of laboratory columns.

They were located a 5-cm intervals for the first 60 cm of depth and at 10 cm for the second 60 cm. The last one was at 135 cm. Samples were removed by inserting a gastight hypodermic needle\* through the septum<sup>##</sup> into the small cavity. Substrate was added periodically to separatory funnels (Fig. 3-2). Flow was automatically regulated with a solenoid valve controlled by an appliance timer. Water-table elevation was controlled with a U-shaped glass connection.

# 3-2 Apparatus for Gas Sampling in Field

Sampling probes similar to those in the laboratory columns were fabricated to obtain gas samples in field studies. Field probes (Fig. 3-3) were of several lengths for sampling at various depths and contained stainlesssteel tubing of larger diameter than the laboratory probes.

Steel rods with a diameter slightly less than the inside diameter of the probes were cut slightly longer than the probe tubing to aid in inserting the sampling probes and to prevent clogging. For sampling, a rod was

- \* Hamilton Company, Inc., Whittier, California. Syringe model 1001LL, needle number KF-72822
- ##Beckman Instruments, Inc., Fullerton, California or The Perkin-Elmer Corporation, Monrovia, California



Fig. 3-3 Probe for collecting gas samples in the field and rod to aid in inserting probe into the soil. placed in the body of a sampling probe and both were inserted vertically into the soil. The rod was removed and the septum and nut were placed on the probe.

Gas was withdrawn by inserting a 30-ml gas-tight hypodermic needle\* through the septum. Samples were placed in culture tubes by water displacement (Fig. 3-4). A cap was then screwed on the tube with the tube tip still submerged. A water seal was maintained by placing the capped tube in the inverted position into a water-filled container.

Samples were later withdrawn in the laboratory from the culture tube by reversing the procedure, using a 1-ml gas-tight hypodermic needle.

## 3-3 Gamma-Radiation Equipment

Soil moisture determinations were obtained by using a 100-millicurie cesium-137 source and measuring gammaradiation attenuation. For these measurements a soil column was placed in a rack that allowed the column to move vertically between source and detector (Fig. 3-5). The shielding was patterned after that of Davidson, Biggar, and Nielsen (33).

The  $Cs^{137}$  source was contained in a hole machined lengthwise through the center of a 4 x 4 x 8-inch lead

\* Hamilton Company, Inc., Whittier, California. Syringe model 1030LL, needle number KF-72822.

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Fig. 3-4 Apparatus to contain gas samples collected in the field.

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Fig. 3-5 Detector, column, and source used to measure moisture by gamma-ray attenuation.

brick. Six 2 x 4 x 8-inch bricks surrounded the brick containing the source, forming an 8 x 8 x 8-inch container. Radiation from the source was collimated before reaching the soil column by passing through openings in two lead bricks. The brick nearest the source was 8 x 8 x 2 inches, with a 0.250-inch diameter hole, 2 inches long, through the center. The second brick was 8 x 4 x 2 inches, with 8-inch side vertical, and contained a 2-inch stainlesssteel tube having inside diameter of 0.125 inches and an outside diameter of 0.250 inches.

The front section of a scintillation detector was inserted in the center of a 4 x 8 x 8-inch lead block, with the front of the thallium-activated sodium iodide crystal flush with the front of the block. The face of the container for the detector was covered with an 8 x 8 x 2-inch plate containing a .875-inch diameter lead plug drilled to accept a 2-inch-long stainless-steel tube with inside diameter of 0.062 inches and outside diameter of 0.083 inches.

Several collimating arrangements were tried before the one described was selected.

Counting equipment to measure gamma-radiation attenuation by the empty columns and to check consistency of packing is shown in Fig. 3-6. The ratemeter signal is for a 10 mv (millivolt) recorder.

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Legend:

- 1. Scintillation detector (Harshaw Type 125/QGX)
- 2. Preamplifier
- Power supply (John Fluke Manufacturing Co, Inc., Model 409 A)
- 4. Single-channel analyzer (Baird Atomic Model 250)
- 5. Scaler (Nuclear-Chicago Model 192 A)
- 6. Ratemeter (Nuclear-Chicago Model 1620 B)
- 7. Recorder (Nuclear-Chicago R-2000 series or
  - Sargent Model SR)

Fig 3-6 Schematic diagram of  $\gamma$ -radiation-counting apparatus

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Because differences in gamma-radiation attenuation during drainage were less than ten percent of attenuation after drainage, it was desirable to expand the portion of the recorder scale showing the differences. A recorder (Sargent Model SR) allowing a variation in range was used in tests involving soil-moisture movements. The scale zero was suppressed by an apparatus that supplied a small voltage to resist the ratemeter signal. The apparatus consisted of a 50,000-ohm potentiometer in parallel with a Heath Company pH-MV Test Unit. The resulting voltage was changed by varying the potentiometer settings or voltage settings on the test unit. The l-mv recorder scale was used when the ratemeter signal was suppressed. <u>3-4 Porous Media</u>

Ottawa quartz sand was one of the porous media used , in the columns. When clean, it had a geometric mean diameter of 0.56 mm, a geometric standard deviation of 1.2, a density of 2.61 gms/cc, and a porosity of 0.35. An abundant microbial culture was grown on the sand by placing the sand in temporary columns and dosing intermittently with settled sewage.

Soil from the top six inches of the Whittier Narrows Test Basin was used in one column. The soil and operating history of this test basin are described by McMichael and McKee (13).

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#### 3-5 Chemical Analyses

Ammonia nitrogen was determined by direct Nesslerization (34). A sample containing less than 0.25 mg of ammonia nitrogen was placed in a 100-ml Nessler tube. To this sample 0.5 ml of 10 percent sodium hexametaphosphate was added to prevent deposition of magnesium and calcium salts upon addition of 2 ml of Nessler reagent (35). Absorbance at 420 m $\mu$  was read and compared with standards.

Kjeldahl-nitrogen determination for organic nitrogen plus ammonia was performed by a micro-analytical method using a mercuric sulfate catalyst with final ammonia determination by Nesslerization. The method is similar to that in <u>Standard Methods</u> (35). The reagent volumes have been adjusted for 50-ml Kjeldahl flasks instead of 800-ml flasks.

Nitrate and nitrite determinations were performed according to <u>Standard Methods</u> (35). The brucine method was used for nitrate and a modified Griess-Ilosvay procedure for nitrite. Extraction from soil water was by a method similar to that of Bremner (36). Twenty-five grams of soil were placed with a saturated calcium sulfate solution into a 250-ml Erlenmeyer flask. Seventy-five ml of calcium sulfate solution were used for tests with Ottawa sand; samples from the Whittier Narrows test basin

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were added to 125 ml. The flask was stoppered and shaken for 10 minutes on a mechanical shaker and the suspension settled. The supernatant was decanted and filtered through a Whatman No. 42 filter paper until the filtrate was clear. The filtrate was then analyzed. To prevent further nitrification the samples were kept in a cold room at 40° F between steps.

The analytical method for chemical oxygen demand was the <u>Standard Methods</u> technique (35).

Glucose and carbohydrate determination was colorimetrically by the anthrone method (37). Except for furfural, no non-carbohydrates have been found to yield positive results with anthrone reagent (37). Of the carbohydrates, the hexoses seem to produce the most intense color. Glucose and fructose produce about equal intensity and galactose yields 54 percent the color of glucose. The contribution from pentoses and uronic acids are negligible at the wavelength (625 m $\mu$  ) and the anthrone concentration used (38). Carbohydrate was determined in soil samples by placing 5-gram samples and 50 ml of 3N H2SO4 in 125-ml Erlenmeyer flasks with mouths covered by marbles to reduce evaporation. The samples were hydrolyzed by placing the flasks on a steam bath for 24 hours. Hot hydrolyzate was passed through medium fritted-disk filters and the residue washed with 50 ml of

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water (38). Samples were diluted and cooled before chemical determination by the anthrone method. Carbohydrate was expressed as "glucose equivalent."

# 3-6 Gas Analysis for Oxygen

Gas analyses for oxygen were made polarographically with an oxygen electrode built by Radiometer of Copenhagen, Denmark. The microelectrode comprises a platinum cathode and silver/silver chloride cathode placed in an electrolytic solution behind a teflon membrane permeable only to gases. The unit is enclosed in a thermostated cell at 38° C. A sodium sulfite solution was used to zero the instrument; air was used to set its range.

Small gas volumes were removed from the columns using the probes described above. A gas-tight syringe needle inserted through the sampling-probe septum withdrew a 1-ml volume which was inserted into the constant temperature cell of the oxygen electrode.

# 3-7 Measurement of Respiration Rates

Respiration rates were determined manometrically with an American Instrument Company Rotary Warburg apparatus. The constant temperature bath was controlled at  $25.35^{\circ} \pm 0.02^{\circ}$  C by a mercury thermoregulator. Manometer fluid was prepared from Spec. D-2930 Meriam Indicating Fluid Concentrate. (0.998 specific gravity at 25° C)

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The center wells of 125-ml BOD Warburg flasks were greased, after which approximately 25 grams of sand were added. One ml of 10-percent KOH and a folded filter paper were placed in each well. Flasks were attached to manometers and placed in the bath, with stopcocks open. After five minutes of shaking, joints were adjusted. The flasks were allowed to equilibrate for 10 to 15 minutes. Manometer fluid was adjusted to reference points, stopcocks were closed, and readings were begun.

Exact sand weights were determined after the measurements were complete and flask constants calculated (39).

Three thermobarometers containing sterile sand were used to correct for temperature or pressure variations. <u>3-8 Soil-Moisture Determination</u>

#### 3-8-1 Gravimetric Determination

A weighed sample was dried for 48 hours at 100 to 110° C and cooled in a desiccator before reweighing. 3-8-2 Moisture-Retention Measurements

A method described by Vomocil (40) was used to determine moisture retention above a water table. The apparatus consisted of a 60-ml Buchner funnel (ultra-fine fritted glass) connected to the bottom of a 50-ml burette by 1/16-inch tygon tubing. After the funnel was boiled to remove air from the disk, the funnel below the disk, the tube, and the burette were filled with water. Sand was placed in the funnel and water added to submerge the sample for 24 hours. Excess water was drained off and loose covers placed on the funnel and burette to reduce evaporation. The burette was lowered in small intervals and the soil water allowed to flow through the disk into the burette. After drainage had ceased, the burette reading and elevation difference between the sand and the water surface in the burette were recorded. The burette was then lowered again and the process repeated several times.

After these measurements, the moisture remaining in the sand was determined as in section 3-8-1.

### 3-8-3 Measuring Soil Moisture by Gamma-Ray Attenuation

Moisture-content measurements are based on  $\mathcal{X}$  -radiation attenuation by soil water.

The attenuation equation for monoenergetic radiation is

 $I=I_{o} exp(-\mu pz)$  (3-1) where I = measured radiation with interference  $I_{o}$  = measured radiation with no interference

> $\nu$  = mass absorption coefficient of absorbing material for the given energy of radiation ( $L^2/M$ )

 $\rho$  = density of material (M/L<sup>3</sup>)

z = sample thickness (L )

Since  $\nu$  varies with radiation energy, equation 3-1 is strictly correct only for radiation of a given energy. This condition was approximated by using an analyzer that measured only radiation with an energy within a narrow range.

The energy spectrum for  $Cs^{137}$  has a radiation peak at 0.66 Mev. (Fig. 3-7). The abscissa does not represent actual energy values of the  $\chi$  photons. The values are, however, proportional. While it would be desirable to measure only those photons with an energy very near the peak, such a limitation is impractical because the position changes slightly with density changes and because instrument amplification drifts slightly. Effects caused by changes in peak position can be minimized by setting upper and lower energy limits symmetrically about the peak so that the slope at the lower setting is zero. For Fig. 3-7 these settings would be 44 and 62 volts.

Equation 3-1 can be rewritten as

$$I = I_{o} e \times p - (\mathcal{N}_{w} p_{w}' z_{w} + \mathcal{N}_{s} p_{s}' z_{s} + \mathcal{N}_{p} \rho_{o} z_{p})$$
(3-2)

in which subscripts refer to water, sand, and plastic. Primes are used for water and sand densities to indicate that these are bulk densities because the substances do not occupy the entire space denoted by z. The equation indicates that moisture content can be determined by measuring changes in attenuation caused by moisture since



Fig. 3-7 Pulse height spectrum for  $Cs^{137}$ .

terms relating to container and sand are constant.

All the terms enclosed by parentheses in equation 3-2 remained constant during the experiment, except for  $\rho'_{\mathcal{W}}$ . Hence, the equation can be rewritten as

 $I = Aexp(-\nu_w P'_w z_w) = A exp(-\nu_w P_w \Theta z_w)$ (3-3) where  $A = I_o exp(-\nu_s P_s z_s + \nu_p P_p z_p)$ 

The value  $\mathcal{M}_w \rho_w z_w$  was obtained by measuring the attenuation of water in a special rectangular container having a 1-1/2-inch hole bored through it. The term  $\Theta$  was calculated from measurements on columns in operation.

#### CHAPTER 4

# EXPERIMENTAL PROCEDURES

# 4-1 Field Experiments

Field studies were conducted at the Whittier Narrows Test Basin used in studies by McMichael and McKee (13). The basin, built on loamy soil previously used for agriculture, is 50 ft by 70 ft in plan and is enclosed by twoft high levees. It is equipped with sampling facilities consisting of a central well with four sampling pans at depths of 2, 4, 6, and 8 ft below the ground surface. The two-ft diameter pans are spaced 60 degrees apart 15 ft from the center of the well and drain to the well through 3/4-inch ID vinyl tubing. Depth to the water table is about nine ft. The basin is covered with a sixinch layer of pea gravel to reduce weed growth.

The Los Angeles County Flood Control District spreads 1.4 to 2.0 ft of activated-sludge effluent from the Whittier Narrows Wastewater Reclamation Plant on the basin once a day, five days a week. Infiltration takes from 10 to 14 hour.

## 4-1-1 Analysis of Soil Water

Soil borings to depths of approximately eight feet were taken on different days beginning 1/2, 5, and 14 hours after the waste had completed its infiltration. A soil boring about one foot long was also taken about six hours after the basin had been spread when the remaining water depth was approximately one foot. Boring operations generally took about one hour. Soil samples were brought to the laboratory (about 11 miles distant) where nitrate in the soil water was determined.

Infiltration took about 10 hours when these studies were made.

# 4-1-2 Analysis of Percolate

Incremental samples of percolate were collected at the Whittier Narrows Test basin for 14 hours after the basin was dosed to a depth of 1.76 ft with effluent. Samples of about 100 ml and 150 ml each were collected from the two-ft pan and 250- and 500-ml samples from the six-ft pan.

The samples were brought to the Whittier Narrows Water Reclamation Plant at intervals of from one to two hours and analyzed immediately for nitrates. Unused portions were then preserved by acidifying and analyzed for Kjeldahl nitrogen later in the week. Sufficient numbers of these samples were analyzed to obtain trends with time for nitrate and Kjeldahl nitrogen.

# 4-1-3 Gas Collection and Analysis

Gas samples were collected at depths ranging from 5.25 inches to 8 ft for times after completion of infiltration of from 5 to 46 hours. Infiltration took about 14 hours when these samples were taken.

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A rod was placed in the body of a field probe (Fig. 3-3) and both were pushed into the soil until the end of the probe was at the desired depth. The rod was then removed and the septum and nut replaced on the probe.

Samples were collected by a 30-ml gas-tight hypodermic needle inserted through the septum. The syringe plunger was pulled out to the desired volume and held for one minute before the needle was withdrawn. The first sample from a probe location, about three times the probe volume, purged the tube and was discarded. About 10 ml gas volumes were then withdrawn from the probe and collected in test tubes.

### 4-2 Laboratory Experiments

# 4-2-1 Filling Columns

The gamma-ray attenuation by the empty columns was measured before filling by taking counts of approximately 100,000 at four locations in each column. Attenuations for individual columns were in close agreement. The largest error found was 1.4 percent from a column mean.

Sand was removed from the temporary columns, mixed thoroughly, air-dried overnight and carefully packed into columns. Soil from the Whittier Narrows Test Basin dried in clumps which were pulverized before being packed. One person poured sand in increments of approximately 50 ml while a second tapped the column at the sand level with a plastic mallet.

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Seven fillings were made during the tests. The uniformity for the first six was checked by measurements of gamma-ray attenuation. Measurements for individual columns were always within 1.6 percent of the mean. 4-2-2 Operation of Columns

Four columns (labelled 1 through 4) were initially filled with 0.56-mm Ottawa sand well seeded with settled sewage and subjected to identical loading patterns but different substrates. Because the solids in wastewaters applied to intermittent sand filters are mostly dissolved, only soluble substrates were first used. Substrate consisted of glucose, ammonium chloride, and salts. Columns 1 and 2 received 400 and 150 mg/1, respectively, of ammonium chloride and no glucose. Column 3 received 200 mg/1 glucose and 42 gm/1 ammonium chloride and Column 4 received , 100 mg/l glucose and 21 mg/l ammonium chloride. Gas analyses taken six weeks after operation had begun indicated little change in oxygen tension in the columns. At that time, substrates to Columns 1 and 3 were strengthened, the medium in Column 2 was changed to soil from the Whittier Narrows Test Basin, and the sand in Column 4 was changed to one composed of about one-half that previously in the column and one-half sand with geometric mean diameter of 0.16 mm and geometric standard deviation of 1.8.

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Substrates and loading patterns for Columns 1 and 3 were changed periodically in an attempt to minimize time required to obtain a measurable oxygen gradient in the column atmosphere. Table 4-1 summarizes the operation of these two columns by listing the substrate concentrations and volumes added at the beginning and at the end of the tests and also the limits of their variations. Infiltration times for dosages of 500 ml were from 2 to 12 hours (Column 1) and 1 to 3 hours (Column 3), depending on the condition of the sand surface. Scarifying the surface down to five centimeters reduced infiltration times toward the lower figures. Column 3 was operated for 4 months. Studies on Column 1 were conducted over about 5-1/2 months.

The Whittier Narrows soil in Column 2 drained slowly after addition of secondary effluent and formed clumps about 1/4 inch in diameter. Only a few air measurements were taken.

Column 4 did not allow sufficient flow with the sand mixture and was repacked with 0.56 mm Ottawa sand. The column was dosed almost daily for 2 months with 500 ml of settled sewage passed through glass wool to reduce suspended matter. Settled sewage was obtained from the Whittier Narrows Water Reclamation Plant and refrigerated until used.

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# TABLE 4-1

		Column 1			Column 3				
		<u>Initial</u>	<u>Final</u>	Range	Initial	Final	Range		
Volume/dose (ml)		250	500		250	500			
Depth/dose (cm)		22	44		22	44			
Doses/day		1	1	1-2	1	2			
Constituents		4							
	Glucose (mg/1)	0	250	0-500	200	500	'		
	NH4C1 (mg/1)	400	100		42	100	42-210		
	<pre>1.0 M Potassium phosphate buffer pH 7 (m1/1)</pre>	1	10		1	10			
	MgSO <sub>4</sub> • 7 H <sub>2</sub> O	20 mg/1	)		а <sup>7</sup>	٠			
	FeCl <sub>3</sub> • 6 H <sub>2</sub> O	0.5 mg/1		same for all tests					
	MnSO <sub>4</sub> · H <sub>2</sub> O	10 mg/1	<pre>same</pre>						
	CaCl <sub>2</sub>	7.5 mg/1		e .					
	Tap Water	50 mg/1	J						

# Summary of Column Operation

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# 4-2-3 Gas Analyses

Gas samples were taken at various times after dosing to determine the variation in oxygen profile during a loading cycle.

The preliminary plan was to obtain only one profile measurement per cycle to avoid distorting air movements. Because measurements were not reproducible from cycle to cycle, owing to changes in surface condition, it was necessary to obtain sequences of measurements during single cycles.

Some probes plugged after a time and could no longer be used for sampling. The probes in Column 1 were unplugged by removing the Swagelok nuts and septa and passing a piano wire through the fine tube.

# 4-2-4 Effluent Analyses

Fifteen-ml increments of effluent were collected to obtain data relating composition and throughput. Analyses included Kjeldahl nitrogen, glucose, nitrate and nitrite nitrogen, and chemical oxygen demand.

# 4-2-5 Measuring Respiration Rates

Following column experiments, Column 3 was disassembled to measure oxygen uptake rates. Five-hundred ml of substrate were added to the surface and the column allowed to drain until more than 475 ml of effluent had been collected. Infiltration took about one hour and drainage about three hours.

The column was then disassembled and soil samples from various depths were placed in Warburg flasks (see Section 3-7) for determination of respiration rates. Oxygen uptake measurements were begun five hours after the column was dosed and continued until 76 hours after addition of nutrient.

#### 4-2-6 Moisture Measurements

Moisture determinations by gamma-attenuation measurements were made at a few depths on Column 3. The column was placed on the rack for radiation measurements about one-half hour before substrate was added and radiation passing through the column was traced with the recorder range at 10 mv. The scale zero was then suppressed until recorder readings were about 10 percent of full scale. The scale was next expanded by changing the recorder range to 1 mv for moisture movements. After addition of substrate to the column, recording was continued until attenuation became almost constant.

Fifteen sand samples were taken after disassembly of Column 3 to obtain moisture content gravimetrically. Moisture-retention measurements were also made on clean sand to determine the effect of bacterial growth on moisture retention. 4-2-7 Chemical Analyses of Sand

Sand from various depths was taken from Columns 3 and 4 after disassembly. Sand from Column 3 was analyzed for chemical oxygen demand, and carbohydrate (glucose equivalent). Samples from Column 4 were analyzed for nitrate and nitrite.

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# CHAPTER 5

#### RESULTS AND DISCUSSION

#### 5-1 Field Experiments

#### 5-1-1 Analysis of Soil Water

Results of analyses for nitrates in soil water at Whittier Narrows are shown in Fig. 5-1. The diagrams indicate nitrate concentrations in soil water about 1/2, 5, and 14 hours after water had infiltrated, and also when the surface was still ponded.

Although the data show considerable scatter, the results during ponding and shortly after infiltration is completed tend to support the hypothesis presented previously that bacterial activity is limited while the surface is ponded and that nitrate content is initially constant with depth after the surface of a soil basin has just drained. The figure also suggests that nitrifying activity progresses downward into the soil as oxygen enters into the soil.

Samples taken 1/2 hour after infiltration show that nitrification has already taken place near the surface and indeed down to one foot or so. After 5 and 14 hours, nitrification has progressed down to about two and four feet, respectively. After 14 hours, the nitrate concentration is about 40 mg/1 for the top four feet and about 15 mg/1 below this depth. Note that the nitrate content of the ponded water was about 6 mg/1.



\*INFLUENT = 6mg/I NO<sub>3</sub>-N

Fig. 5-1 Nitrate profiles in soil water at Whittier Narrows Test Basin. (Ten-hour infiltration time)

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No attempt was made to measure ammonium or other forms of nitrogen with minus-three oxidation state. These tests were to measure only dissolved substances containing nitrogen. Extraction as used in determining nitrate in the soil water would have removed ammonium weakly adsorbed to soil surfaces and caused nitrogenous matter to slough off during shaking.

The model presented in Section 2-3 proposed that nitrate below four feet is low because the liquid passed through the biologically active zone when oxygen was low and therefore was never nitrified. An alternative hypothesis for the decrease in nitrate below four feet could be that denitrification has taken place. However, data from the report by McMichael and McKee (13) for samples at six and eight feet indicate no decrease in total nitrogen. Discussion in section 5-1-3 presents further evidence against denitrification.

# 5-1-2 Analysis of Percolate

Incremental samples collected from the sampling pans did not demonstrate the nitrate wave sketched in Fig. 2-3. Kjeldahl-nitrogen concentrations for samples from the pan at a depth of two ft remained at about 0.5 mg/l through the entire sampling period; from the six-ft pan at about 1.8 to 2.0 mg/l. Concentrations of nitrate nitrogen for effluent taken from the two-ft pan varied from about 24

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to 32 mg/l after the initial few hundred ml in which nitrate was somewhat lower. Effluent from the six-ft pan was not analyzed for nitrate.

Using a sodium chloride tracer on the same basin, McMichael (41) showed that there was a 38-hour delay between the time tracer was added to the ponded water and the time it was first observed at the two-ft pan. The delay was 28 hours at the six-ft pan. In addition, the quantities of water collected were only 8 and 17 percent of those expected and the flow-through characteristics of the four sampling pans were markedly different. These results imply that the water collected at the pans is not representative of newly percolated water.

Water perched in the capillary fringe above pans impedes flow into the pans and diverts flow around them. Thus, the composition of water collected by sampling pans does not represent that of water percolating nearby, but rather that of water held in the fringe for a day or more.

Problems caused by perched water can be avoided by using porous ceramic cups through which soil solution is extracted by applying tension to the cups with a vacuum pump or hanging water column (42, 43). However, a better way to obtain a continuous sample of percolate that is truly representative seems to be by means of column experiments, in which case the entire percolate is collected.

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In addition, the column experiments can be used to evaluate the proposed model.

### 5-1-3 Gas Collection and Analysis

The data for oxygen content of gas samples taken at various depths at Whittier Narrows for times of 5 to 46 hours after completion of infiltration are plotted in Fig. 5-2. Each point is the average of two samples taken at a probe location. Samples with differences of greater than 10 mm mercury (.013 atmospheres) were not plotted. Samples could not be obtained from some locations, possibly because the probes became plugged with mud as a partial • vacuum was applied by withdrawing gas.

The data show no trend with time but are scattered within the envelopes in Fig. 5-2. A small part of the scatter may be due to leakage around the septa, but most irregularity was probably caused by the non-uniformity of the soil porosity. In sampling of this type, the sample is drawn from the larger pores about the probe. Hence any cracks about the bottom of the probe would have contributed a disproportionate part of the gas volume and the sample withdrawn would not have given a true indication of the oxygen content at the sampling point.

The results indicate that oxygen did not penetrate even to two feet at the test basin during these tests.

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Fig. 5-2 Measurements of partial pressure of oxygen in soil atmosphere at Whittier Narrows Test Basin.

The gas samples were taken about nine months after the cores for analysis of soil water were taken, during which interval the infiltration time increased from 10 to 14 hours. A new core was thus taken to determine whether the nitrate profile had changed. The results for a core taken about 11 hours after completion of infiltration (Fig. 5-3) show that the active zone for nitrification has decreased to less than two feet, roughly the depth where anaerobic conditions begin (Fig. 5-2).

In section 5-1-1, denitrification was presented as a remote possibility for the decrease in nitrate content beyond the first few feet of an intermittent sand filter. Denitrification could occur, of course, only if there had been prior nitrification. Fig. 5-1 shows that nitrate content was low for all depths immediately following infiltration. Data in Fig. 5-2 show that oxygen did not penetrate more than two feet into the soil when the data for Fig. 5-3 were collected, so that nitrification could not have taken place beyond two feet at that time (or beyond four feet when the data for Fig. 5-1 were obtained). Since nitrification was absent, denitrification could not have caused the large decrease in nitrate concentration.

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5-3 Nitrate profile in soil water at Whitter Narrows Test Basin llhours after completion of infiltration. (Fourteen-hour infiltration time.)

### 5-2 Laboratory Experiments

# 5-2-1 Gas Analyses

Oxygen profiles in the laboratory columns were not reproducible from cycle to cycle, but depended on the condition of the sand near the surface, where bacterial growth was observed to be much more dense than elsewhere. The growth reduced infiltration rates and effective gas diffusivity. Because short infiltration times were wanted to allow atmospheric oxygen to be available for a large part of the cycle, the column surface was scarified from time to time to increase infiltration rate. Besides increasing infiltration rate, scarifying also allowed oxygen to penetrate to a greater depth.

The results of oxygen analyses for gas samples taken during two cycles of Column 3 are shown in Fig. 5-4. The samples for each cycle were taken at 7.9, 24.5, 49.3, and 71.3 hours after substrate was added. As noted, the profiles are not reproducible, but the two sets of data are similar in shape and trend. Because the curves for each set do not vary greatly over a time span of more than 63 hours, the assumption that the system reaches a quasisteady state after some time seems tenable. Such reasoning would be poor if the curves in a cycle differed greatly. With rapid changes, oxygen concentrations would depend not only on conditions (such as effective diffusivity

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Fig. 5-4 Profiles of oxygen partial pressure: in Column 3 during two series for times after dosing of 7.9, 24.5, 49.3, and 71.3 hours.

and bacterial-respiration rates) at a given time, but also on past conditions. Since quasi-steady state develops under the conditions of this experiment, the rate of oxygen addition by diffusion is equaled at each depth by the rate of deoxygenation from bacterial respiration.

Partial pressures of oxygen for samples taken about one hour after Column 1 was ponded with 500 ml of a 500-mg/l glucose solution and while the column was still ponded are presented in Table 5-1. The partial pressures, all less than 1.3 percent of those in the atmosphere (except for that at 60 cm), show that the column was essentially anaerobic during ponding. Some of the error can be attributed to contamination of the sample during withdrawal and analysis. One hour was about the minimum duration of ponding for Columns 1 and 3 after they had been dosed intermittently for about two months. These measurements therefore indicate that the atmosphere in the columns was devoid of oxygen just as infiltration was terminating.

Another series of gas samples was taken from Column 3 to study the distribution of oxygen before the quasi-steady state had developed. The data (Fig. 5-5) indicate that this condition had not been attained at about 3.5 hours after substrate addition. This time corresponded to that when drainage (infiltration and percolation) had essentially ceased. Measurements at 12 and 23 hours after

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# TABLE 5-1

Partial Pressures of Oxygen in a Ponded Column One Hour After Substrate Was Ponded

Depth (cm)		<u>Partial Pressure (atm)</u>
10		*
15		*
20		.0015
30	·	.0019
40		.0015
50		.0025
60		.0041

\* Water rather than air was withdrawn by the

sampling procedure

substrate addition show that the system was at a quasisteady state. These measurements together with those of Fig. 5-4 show that the quasi-steady state was attained between 3.5 and 7.9 hours after substrate addition or between 2.5 and 6.9 hours after termination of infiltration.

Oxygen partial pressures were substantially higher at 3.5 hours than later because drainage allowed oxygen to enter the system by convection in addition to diffusion. At this time, the system could not be characterized by a quasi-

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Fig. 5-5 Profiles of oxygen partial pressure in Column 3 for times after dosing of 3.5, 12, and 23 hours.

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•,

steady state because the oxygen profile depended on the past history rather than just on respiration rates and gas porosity at 3.5 hours. As drainage rates decreased, the oxygen partial pressures decreased to a quasi steady-state oxygen profile and then increased slowly with time.

#### 5-2-2 Respiration Rates

Data calculated from measurements of respiration rates in Column 3 are shown in Fig. 5-6. The recorded data were first plotted for each Warburg flask on graphs such as Fig. 5-7 and slopes in terms of mm/hour obtained for times corresponding to those for the oxygen analyses (Fig. 5-2). Bacterial-respiration rates were then calculated by multiplying the slopes by the flask constant per gram of dry soil.

Most of the aerobic bacterial activity occurred near , the surface, decreasing by more than an order of magnitude in about 40 cm and remaining roughly constant from 40 to 70 cm. Trends in Fig. 5-6 show that oxygen-uptake rates continuously decreased during the experiment.

It has been shown (44,45) that the rate of oxygen uptake for bacterial systems increases during the growth phase and that the beginning of decreased oxygen-uptake rate corresponds to the end of the growth phase. The decrease of oxygen uptake rates with time indicates that many microorganisms were already respiring more quickly

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than they were assimilating substrate when the Warburg measurements began about five hours after addition of substrate. Therefore, easily degradable substrate was no longer readily available to most bacteria.

#### 5-2-3 Moisture Retention Measurements

A comparison of moisture profiles for 0.56 mm Ottawa sand when clean and after use in Column 3 is shown in Fig. 5-8. The data for clean sand were obtained with a frittedglass Buchner funnel (Section 3-8-2). It should be noted that the ordinates of points plotted for the clean sand are distances above a water table and that the ordinate for the uppermost point is not a negative depth. The data for sand from Column 3 were obtained gravimetrically after disassembly. The latter points represent the condition about three hours after addition of substrate, when moisture movements had almost ceased.

Bacterial growth caused a large increase in moisture retention for the greater part of the column. The change in moisture retention near the surface is important because it diminishes the effective diffusivity for gases by decreasing the porosity.

### 5-2-4 Measurements of Redistribution of Moisture

Attenuation of a 1.5-inch diameter cylinder of water was determined from gamma-radiation measurements on an acrylic-plastic container with the same cross-section as

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Fig. 5-8 Moisture profiles after drainage of clean Ottawa sand and Ottawa sand after use in Column 3.

the columns. The term  $\mathcal{N}_{w}Z_{w}\mathcal{P}_{w}$  in equation 3-3 for a 1.5-inch cylinder of water was found to be 0.314, corresponding to a mass adsorption coefficient ( $\mathcal{N}_{w}$ ) of 0.0827 cm<sup>2</sup>/gm for a water thickness of 1.5 inches. The value is within the range of 0.0815 to 0.0841 determined by others (33, 46).

Moisture changes during percolation of liquid were determined by measurements taken during four cycles. Different locations were selected each time. The results in Fig. 5-9 show no trend with depth. Although "noise" in the instrumentation caused some inaccuracy , the lack of an obvious drainage pattern can be attributed to the differences in the conditions near the surface, which conditions caused infiltration rates to vary considerably from day to day. The surface was scarified at intervals to maintain high infiltration rates. Even though the results are not consistent, some conclusions can still be drawn from the plots.

Information in Figs. 5-8 and 5-9 shows that water content before new infiltration was about 27 percent of saturation and increased to about 57 percent of saturation during infiltration for depths from 10 to 130 cm. Therefore, gas volume decreased from 73 to 43 percent of total pore volume in this distance, a volume decrease of about 160 ml. As a check, gas volume expelled at the column

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Fig. 5-9 Increase in soil moisture in Column 3 after addition of 44 cm of wastewater. Distances indicated near curves are depths from surface. Base line is moisture before dosing.

outlet was measured by water displacement and found to be about 140 ml. The total gas volume of the drained column can be calculated to be about 380 ml. Therefore, the water advancing in the column replaced only about 40 percent of the total gas volume.

For each case drawn in Fig. 5-9, the water content begins to decrease at the same time as infiltration is completed. The flow pattern was thus similar to that obtained by Biswas, <u>et al</u> (14) for redistribution in fine-textured soils, in which flow was saturated only at the surface and moisture contents decreased as ponding ceased (See Fig. 2-1b). The Ottawa sand used in these studies was not fine textured, but microorganisms and associated products in the column sufficiently reduced permeability at the surface so that the flow was unsaturated through most of the column. ', Similar behavior is to be expected in intermittent sand filters using other soils because growths should cause the minimum permeability to occur near the surface.

# 5-2-5 Analysis of Oxygen Transfer

The sets of curves in Fig. 5-4 for the two test series do not coincide because of differences in the surface condition of the column. The sand surface was scratched with a thin rod before substrate was added at the beginning of each series. The resulting condition of the sand matrix near the surface after the wastes had infiltrated was appar-

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ently different in the two tests causing a change in total porosity, moisture retention, and therefore in effective diffusivity. Some of the difference in the oxygen profiles can be attributed to changes in respiration rates in the interval between the two sequences, but this difference is minor because respiration rate should change only slightly over a period of a few weeks after columns have been operating for several months.

In order to test the quasi steady-state model for oxygen transfer in intermittent sand filtration on the basis of measured parameters, equation 2-13 was used:

$$-\frac{d}{dz}\left(c\mathcal{D}\frac{dx_{A}}{dz}\right) = R_{A}$$
(2-13)

During a series of measurements, the oxygen-concentration profile and the respiration rates changed, but the effective diffusivity at a given depth should have remained constant once moisture movements ended. The applicability of equation 2-13 to the problem can be determined, therefore, by using measured data on oxygen concentrations and respiration rates, and calculating the effective diffusivity. If the effective diffusivity at any given depth does not change with time, the model should be satisfactory.

To solve for effective diffusivity, equation 2-13 was integrated once and rearranged to yield

$$\mathcal{D}(z) = \frac{\int_{z}^{L} R_{A}(\xi) d\xi}{c \frac{dx_{A}}{dz}}$$
(2-18)

Diffusivities during the two series in Fig. 5-4 were calculated by inserting into equation 2-18 respiration rates from Fig. 5-6 and slopes of the oxygen profiles in Fig. 5-4.

The distance L at which the partial pressure of oxygen becomes zero was first obtained from each of the eight curves in Fig. 5-4. The integral  $\int_{z}^{L} \mathcal{R}_{A}(\xi) d\xi$  was next calculated numerically with a digital-computer subroutine for integrating a plot function.

Slopes  $dx_A/dz$  of the curves fitted to the points in Fig. 5-4 were measured for the distances in the subroutine output, and the diffusivities calculated from equation 2-18. This process was repeated for each of the eight curves in Fig. 5-4 with the appropriate respiration rates from Fig. 5-6.

Calculated diffusivities for the four times in each of the two series are plotted in Fig. 5-10, with a curve drawn to fit the points. In both series, diffusivities are lower at the surface than elsewhere and increase with depth before becoming roughly constant. Diffusivities are low at the surface where bacteria reduce gas porosity because of a decrease of total pore volume by their bulk and their waste products and because of increased moisture retention. Fig. 5-10 indicates that bacterial growth can reduce the effective diffusivity at the surface to between 12 and 37 percent of that at about fifteen to thirty cm. The calculated diffusivities show that changing the sand structure near the

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Fig. 5-10 Effective diffusivity calculated from quasi steady-state model (equation 2-13) using measured respiration rates (Fig. 5-6) and oxygen profiles (Fig. 5-4). Solid lines are fitted to data from model. Crosses fitted by dashed lines are calculated from Currie (27) data using moisture data from Fig. 5-8 and  $\mathcal{D}_{\circ} = 806 \text{ cm}^2/\text{hr}.$ 

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surface by scarification influenced the moisture retention (and, therefore, effective diffusivity) for a depth much greater than that of scarification. Effective diffusivities in Series A did not exceed about 30 cm<sup>2</sup>/hour whereas in Series B they reached about 50 cm<sup>2</sup>/hour.

A model is realistic only if the parameters calculated by it can be compared with at least some agreement to values calculated by an independent method. Diffusivities calculated using formulas taken from data plotted by Currie (27) can be used as the independent check (See Section 2-4). These diffusivities, with moisture contents from Fig. 5-8 and  $\mathcal{D}_{\circ} = 806 \text{ cm}^2/\text{hour}$ , are plotted as crosses fitted with dashed lines in Fig. 5-10. The results indicate that the diffusivities calculated by the model agree within a factor of two with those calculated on the basis of gas porosities.

The diffusivities calculated from respiration rates and oxygen profiles are all close to the fitted curve. Theoretically, there should be no scatter of the points at any depth during a series if moisture movements have stopped, but inaccuracies result from errors in measuring the slope of the oxygen profile and in determining oxygen uptake in the Warburg flasks. To determine if the scatter is caused by experimental error and not by error in the model, oxygen profiles were calculated using diffusivities from the curves in Fig. 5-10 and respiration rates from Fig. 5-6. The oxygen profiles were calculated using equation 2-19

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$$x_{A} = x_{A0} + \frac{1}{c} \int_{0}^{z} \frac{\int_{z}^{L} R_{A}(\xi) d\xi}{D(\xi)}$$
(2-19)

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which was solved numerically with the use of the IBM 7094 digital computer.

Because data for  $R_A$  were available only to 70 cm depth and it was felt that values of  $R_A$  might be needed for greater depths, estimates of  $R_A$  were added to the program for these depths. The estimates were 1.7, 1.5, 1.4, and 1.2  $\mu$ 1/gm/hr for times after dosing of 7.9, 24.5, 49.3, and 71.3 hours.

The same procedure was followed for each time to calculate variations of oxygen partial pressure with depth. Values of  $\int_{0}^{z} R_{A}(\xi) d\xi$  for z ranging from 0 to 70 cm were calculated with a subroutine for integrating a plot function. Values of  $\int_{0}^{z} R_{A}(\xi) d\xi$  for z greater than 70 cm were obtained by adding the area contributed by the estimated activity to that calculated at 70 cm.

The two boundary conditions for equation 2-13 were that  $x_A = x_{A0}$  at the surface and that  $dx_A/dz = 0$  at a depth L where aerobic bacterial respiration becomes zero because oxygen is absent. Because it was required that  $x_A$  also be zero at L this distance had to be estimated by trial and error. The scheme was to choose a trial of L of 10 cm and to calculate  $x_A$  from 0 to L. If  $x_A$  at L was positive, the next estimate of L was taken as 10 cm more and the process repeated until  $x_A$  at L was negative. A new L was then

chosen 9 cm less than the last value and  $x_A$  magnitudes calculated. A positive  $x_A$  at L caused a 1.0-cm increase in the trial L until  $x_A$  was negative at L. The depth L was reduced by 0.9 cm and then increased in 0.1-cm steps until  $x_A$  at L became negative. The first trial L for the next time for which experimental values were available was the last calculated L.

The value of  $\int_{0}^{t} R_{A}(\xi) d\xi$  for the trial L was estimated by second-order interpolation. Diffusivities for the values of z used in the integration subroutine were estimated by interpolation.

The terms

$$\frac{\int_{z}^{L} R_{A}(\xi) d\xi}{\mathcal{D}(\xi)}$$

were calculated for the z's from the integration subroutine ; and

$$\int_{a}^{2} \frac{\int_{z}^{L} R_{A}(\xi) d\xi}{\mathcal{D}(\xi)} d\xi$$

obtained from the same subroutine. Oxygen partial pressures from 0 to L were then calculated from equation 2-19.

The results of the calculations (Fig. 5-11) except for 7.9 hours after dosing agree quite well with the oxygen concentrations measured in Column 3. They support the assumption of a quasi-steady state for the description of the oxygen profile and show that the change in respiration rates



Fig. 5-11 Comparison of oxygen partial pressures calculated by quasi steady-state model with measured data.

is sufficient to alter the oxygen profile. The discrepancy at 7.9 hours after dosing can be attributed, at least in part, to the different test conditions. In the laboratory columns, oxygen does not reach some bacteria, especially near the point where two sand particles touch, because these bacteria are covered by other bacteria. Sanders (47) estimated that bacteria covered by more than two layers of other bacteria are limited by a lack of oxygen. The calculated curves are based on respiration rates obtained with a Warburg apparatus, in which sand particles tend to be separated. Hence, some bacteria that were covered by other layers in the sand columns became exposed to atmospheric oxygen when sand particles were separated and exerted a high initial oxygen-uptake rate.

No estimates of  $\mathcal{D}$  and  $R_A$  are presently available for intermittent sand filters. Both values depend on the bacterial growth attained in the system, which growth in turn depends on many parameters, including composition and strength of wastewater, depth and frequency of ponding, and soil type. Since all these factors can be selected in designing and operating intermittent sand filters, their effect on  $\mathcal{D}$  and  $R_A$  deserves more study.

5-2-6 Effluent Analyses

Effluent analyses on Columns 1 and 3 which were dosed with solutions of glucose, ammonium chloride, and salts

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varied considerably during the study. Both columns initially formed nitrates, but other forms of bacteria apparently overgrew the nitrifying bacteria during the study because nitrate was not present in the effluent after four months of operation. It is not implied that glucose by its presence inhibited nitrification, although some organic compounds do inhibit. Glucose in concentrations less than 0.3M does not poison nitrifiers (48) but heterotrophic bacteria using it as substrate can assimilate all the ammonia present if initial ammonia concentration is low, so that no ammonia is available for nitrifying bacteria (49). Because ammonium was always present in the column effluent in this study, ammonium was available to nitrifying bacteria. The generation times for heterotrophic organisms are so much less than those of the nitrifiers (30 minutes vs 31 hours) that heterotrophic bacteria covered the nitrifying bacteria and prevented oxygen from reaching them.

Effluents from Columns 1 and 3 were collected during several cycles to determine the glucose present. Glucose removal was complete for the entire effluent collected during most cycles, but glucose was present in a few effluents. The latter results are of interest because they show the variation in removal with throughput when removal is not complete. Fig. 5-12 shows trends obtained

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Fig. 5-12 Variation of glucose concentration with throughput from Column 3.

during cycles for which glucose was measured in the effluent. The effluent glucose was low at the beginning of drainage when the liquid that had been in the column for most of a cycle was being displaced. Glucose concentration then increased in the effluent for liquid that passed through the column while the column was anaerobic. The decrease for Column 1 after 420 ml indicates the effect of a reduction of flow rate, thus allowing more time for bacteria to metabolize glucose.

Considerable amounts of glucose were removed under anaerobic conditions because the effluent glucose concentrations in Fig. 5-12 are always much less than the influent concentration. It is generally assumed that adsorption of glucose on organic matter from bacterial growth is negligible compared to metabolism as a mechanism for rapid substrate removal in aerobic systems. This assumption is based on studies of activated sludge, such as those by Gaudy and Engelbrecht (44) and by Krishnan and Gaudy (50). Jeris and Cardenas (51) studied aerobic and anaerobic biodegradation of glucose in laboratory-scale units to which a daily dose of glucose was added. Results showed a zero-order reaction for both The results for the aerobic system when extrasystems. polated back to zero time agreed with the amount of glucose added within about three percent. Extrapolation for two

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anaerobic systems resulted in values at time zero that were 7 to 24 percent less than the glucose added. The disagreement in the latter cases may have been caused by adsorption of glucose. Thus, it appears from the work by Jeris and Cardenas (51) that both adsorption and biodegradation can be responsible for rapid removal of glucose in anaerobic systems. Applied to this study, their work suggests that glucose was removed from the percolating liquid by adsorption and degradation.

Because nitrogenous material is an important fraction of the components of wastewaters and because no nitrate was formed in Columns 1 and 3, a column (Column 4) was refilled with 0.56 mm Ottawa sand and dosed with settled sewage to study nitrification. Gas analyses indicated that Column 4 remained aerobic down to the top of the capillary fringe. At Whittier Narrows, nitrification is prevented at some depths because the soil atmosphere lacks oxygen; in Column 4, nitrification occurred down to the top of the capillary fringe and to a certain extent within the fringe, where lack of oxygen also diminished nitrification.

Effluent from Column 4 was analyzed for Kjeldahl, nitrate, and nitrite nitrogen and for COD to determine their variation with throughput. The test for Kjeldahl nitrogen measures the concentration of nitrogen having

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minus-three valence. The bacteria in the system served to oxidize the nitrogen to nitrite and nitrate, thereby lessening the Kjeldahl nitrogen present. Tests for the specific carbonaceous components in wastewaters would be preferred to the COD determination which is to measure the portion of organic matter that can be oxidized by a strong oxidant. Nevertheless, because of the large number of carbonaceous components in wastewaters, the COD test was used as an expedient.

The nitrogen measurements in Fig. 5-13 show large variation in effluent quality with throughput. The first increments collected, low in nitrate and high in Kjeldahl nitrogen, came from the capillary fringe where lack of oxygen limited nitrification. The nitrate peak and accompanying Kjeldahl-nitrogen valley correspond to . liquid that had been in the aerobic portion of the column for almost a day. The position of the nitrate peak ahead of the total moisture retained in the column and the low nitrate concentration for larger throughput indicate that little nitrification occurred while liquid was draining. The decrease in Kjeldahl nitrogen during drainage was caused by filtration and adsorption of suspended and dissolved nitrogenous matter. Dispersion accounts for the nitrate nitrogen after drainage of a volume equal to the moisture in the column. Note that the ordinate values for

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Fig. 5-13 Variation of nitrite, nitrate, and Kjeldahl nitrogen with throughput from Column 4.

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nitrite have been multiplied by 100. The actual values are so small, all less than 0.15 mg/1, that they can be neglected in arriving at a nitrogen balance. No obvious reason has been found to explain the position of the nitrite peak after the nitrate peak, rather than at the same value of throughput.

The low COD for initial throughput (Fig. 5-14) results from displacement of liquid that was in the column for most of the cycle. COD content then rises with throughput because COD removal is limited during drainage. COD removal (Fig. 5-14) during column drainage was probably due mostly to filtration and adsorption rather than biodegradation because infiltration took only ten minutes. The settled sewage, even after passing through glass wool, still contained colloidal and particulate matter, which could be removed by the sand media. The decrease in COD after 380-ml throughput illustrates the improvement in COD removal when the percolate drains slowly and allows the wastes to remain in the column for a longer time.

Results from Column 4 in Figs. 5-13 and 5-14 can be compared with what would be expected from a spreading basin used for ground-water recharge. In a spreading basin, the wastewater passes through an aerobic portion a few feet deep and then trickles through an anaerobic zone

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Fig. 5-14 Variation of chemical oxygen demand with throughput from Column 4.

to the water table. In the anaerobic zone, no further nitrification takes place, but some carbonaceous material may be removed from the percolate. Column 4 was similar except that the water table was brought up to the aerobic zone, and the percolate was collected just below the water table. Once collected, the percolate was not stabilized further, if it was immediately refrigerated. The similarity indicates that the variation of nitrate with throughput for a spreading basin should be comparable to the variation presented in Fig. 5-13 for a laboratory column. The COD reaching the water table below a spreading basin should be less than in the laboratory because bacterial activity in the anaerobic zone will diminish the carbonaceous matter.

### 5-2-7 Chemical Analyses of Sand Samples

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Sand from Column 3 was analyzed for COD and for carbohydrate (glucose equivalent). The results (Fig. 5-15) along with those on bacterial respiration (Fig. 5-6) can be taken as indices of biological growth. They indicate that most of the bacterial growth occurs near the surface. This fact has been observed by many investigators using wastewaters, which contain colloidal and particulate matter as well as dissolved material. The measurements on Column 3 show that nutrient consisting of dissolved substrates only will also promote the same growth pattern.

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Among the conditions affecting the distribution of bacterial growth in intermittent sand filtration, reduced oxygen concentration with increasing depth is most important, although other factors modify the growth pattern. Denser growth in the upper section of the sand column, whether caused at first by oxygen limitations at greater depth or by complete substrate removal in the upper section will decrease the permeability near the surface. Takagi (52) has shown that ponding water on a material of sufficiently low permeability above one of higher permeability will produce uniformly unsaturated flow in the lower section with the transition occurring within the region of low permeability. Once ponding ceases, the moisture probably drains as in Fig. 2-lb, with greater moisture content near the surface than elsewhere. This pattern allows , substrate to be available for a longer time to organisms near the top, if all the easily oxidized substrate is stabilized during a cycle. Operation continued over many cycles further reduces the permeability near the surface and produces moisture retention as in Fig. 5-8. Because substrate is available near the surface for a longer portion of a cycle, more bacteria will be able to grow there.

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Results (Fig. 5-16) for nitrate and nitrite extracted from sand in Column 4 twenty-four hours after substrate addition show similar trends for both anions, with nitrate concentrations roughly one hundred times nitrite concentrations. On Fig. 5-17 the nitrate is replotted in terms of  $\nu$  g nitrate nitrogen per gram of dry sand. The increases in total nitrate at 0 to 10 cm and 120 to 140 cm correlate with increases in moisture content (Fig. 5-17). A larger moisture content, accompanied by larger total Kjeldahl nitrogen, allows substrate to be available to nitrifiers for a longer time during the cycle. Nitrifying organisms between 10 and 120 cm probably used up all of the readily available substrate. The decrease in total nitrate from 140 to 150 cm is attributable to the lack of oxygen. The increase between 0 and 10 cm above that from . increased substrate volume was caused by availability of colloidal and particulate matter, which was removed by filtration.

The nitrate analyses on the soil extract indicate that 6900  $\mu$  g of nitrate nitrogen were produced during a cycle. However, the effluent analyses (Fig. 5-13) suggest that only 6140  $\mu$  g were produced. Denitrification does not seem to be the cause of disagreement because effluent analyses for nitrate and Kjeldahl nitrogen show a nitrogen balance (within two percent). Instead, a systematic error in one of the analyses may be the cause.

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Fig. 5-16 Profiles of nitrate and nitrite concentrations in soil moisture in Column 4.



Fig. 5-17 Moisture profile after drainage of sand in Column 4 and profile of nitrate content per gram dry sand.

#### CHAPTER 6

### SUMMARY AND CONCLUSIONS

#### 6-1 Rationale of this Research

Although almost a hundred years of experience with intermittent sand filtration of wastewater had shown clearly that the system must be kept aerobic to prevent clogging, very little work had been done to study oxygen relationships in the system. For example, an extensive review of the literature did not uncover a single measurement of oxygen concentrations in the soil atmosphere for media used for treatment of sewage by intermittent percolation. The increasing use of spreading basins for tertiary treatment and ground-water recharge makes research on the oxygen relationships in soil systems pertinent inasmuch as oxygen availability may limit the usefulness of the method.

A simplified description of the various processes that occur during intermittent sand filtration was also needed because an understanding of the soil system as a whole is necessary to comprehend transfer and bacterial utilization of oxygen. A simplified description of the processes is useful by itself to explain variations in the quality of effluent from an intermittent sand filter.

## 6-2 Summary of Processes

#### 6-2-1 Spreading Basin Operation

In practice, effluent from a primary or secondary wastewater treatment plant is spread on a basin of sand or soil and the area is allowed to drain until no liquid is ponded on the surface. The basin is then "rested" before further application of treated wastes to allow oxygen from the atmosphere to diffuse into the soil. Total cycle times (ponded plus resting) vary from about 4 hours to three weeks or more, with resting times constituting half or more of the total cycle.

#### 6-2-2 Description of Processes

When effluent from a secondary treatment plant is applied to the surface of a spreading basin, pollutants are removed from the incoming liquid by adsorption on soil surfaces, by diffusion into stagnant zones, and by biological assimilation and synthesis. Concurrently, stabilized substances are returned to the percolate and move with it.

Biological activity within the sand or soil is limited while the surface of a spreading basin is ponded. Lack of oxygen limits the activity of aerobic organisms such as fungi and nitrifying bacteria. Anaerobic bacteria can degrade only portions of the available substrate because the liquid percolates through the soil too rapidly. Some components, such as alkyl benzene sulfonates, may not be entirely removed from the percolate because the adsorbing surfaces become saturated.

After infiltration has ceased, oxygen reenters into the soil and aerobic bacterial activity progresses toward greater depths. The depth of aerobic bacterial activity is limited, however, because oxygen is depleted by biochemical utilization as air diffuses downward. Hence, some depths are always anaerobic. Consequently, not all percolate passes through the system with the same degree of treatment. Parts of the effluent that go beyond the maximum depth where oxygen is ever present receive no further aerobic treatment. It is possible, therefore, for percolate to show large variations with respect to the concentration of some contaminants, and some effluent portions show little change in these contaminants in the influent.

Nitrate can be used to indicate the extent of aerobic activity in the soil and to demonstrate the effects of displacement of pellicular water when wastewater is applied to the surface of a spreading basin. Because nitrification rates are low while the surface is ponded, the nitrate concentration in the soil water is roughly constant for some depth into the ground just as infiltration is completed. Nitrate is first formed near the surface and is formed at greater depths as oxygen becomes available. Nitrate cannot be produced, however, at depths where oxygen is absent.

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It serves therefore to indicate the maximum depth to which oxygen penetrates.

Displacement of the nitrate formed during one cycle by newly applied wastewater is hypothesized to cause the percolate to have a nitrate wave (Fig. 2-3).

### 6-2-3 Oxygen Transport

The complexity of redistribution of moisture during intermittent percolation and the effect of such moisture redistribution on the movement of gas make any attempt at mathematically describing the oxygen concentration while moisture is still draining most difficult, if not impossible. It was observed, however, that after infiltration is completed, there developes a quasi-steady state, one in which the oxygen concentration does not depend on the past history of the media. This condition exists in the soil for several days after infiltration has stopped.

The differential equation describing the quasi-steady state is

$$-\frac{d}{dz}\left(\frac{l}{l-x_{A}+qX_{A}} \quad c \mathcal{D} \quad \frac{dX_{A}}{dz}\right) = R_{A}$$
(2-15)

for which notation is presented in Chapter 2. Because respiratory quotients (q) for intermittent sand filtration are not known, but probably lie between zero and one, equation 2-15 was solved for a simple model first with q equal to zero and then to one. The two solutions were reasonably close. It was thus decided to use equation 2-15 with a respiratory quotient equal to one to analyze data. 6-3 Principal Results

1. Analyses were conducted on soil samples from the Whittier Narrows Test Basin to determine the nitrate concentration in the soil water. As predicted by the foregoing discussion, the data show that only slight nitrification occurs while the basin is ponded and that the nitrate content in the soil moisture is roughly constant for some depth immediately after infiltration stops. Nitrification begins near the surface after infiltration ends and progresses downward as oxygen enters into the soil. The nitrate profile just before wastewater is added again to the surface shows high nitrate concentration for the first few feet and then a sharp drop in nitrate concentration where oxygen failed to penetrate and hence where no increase in nitrification occurred after infiltration.

2. Oxygen concentrations in gas samples taken from the soil atmosphere at the basin showed no trend with time but indicated that oxygen penetrated only to about two feet or less for times from 5 to 46 hours after completion of infiltration. Measurements taken at the same time to determine nitrate in soil water agreed with the oxygen measurements by showing that nitrification also took place only within the top two feet of the basin. 3. Oxygen profiles measured in laboratory columns dosed intermittently with a synthetic mixture showed only slight changes over more than 63 hours and could be described at sufficiently long times after substrate addition by a quasi steady-state equation for oxygen transport. The laboratory oxygen profiles and respiration rates obtained with a Warburg respirometer were used to calculate diffusivities from equation 2-18:

$$\mathcal{D}(z) = \frac{\int_{z}^{z} R_{A}(\xi) d\xi}{c \frac{dx_{A}}{dz}}$$
(2-18)

The diffusivities were almost independent of time and were used to calculate new oxygen profiles which agreed quite well with the experimental profiles. Results indicated that scarifying the surface of the media influenced effective diffusivity for a depth much greater than that of scarification.

4. Data from gravimetric moisture determinations and from moisture determinations by gamma-radiation attentuation indicated that flow was always unsaturated throughout most of the column. Microorganisms and associated products at or near the surface of the column limited infiltration rates, thereby causing the lower areas to be unsaturated.

5. Incremental effluent samples were collected from a column dosed with glucose, ammonium chloride, and salts. Analyses for glucose in the effluent showed that glucose removal was almost complete for the initial throughput in a cycle. Glucose concentrations, although increasing during further drainage, never approached the influent concentrations. Since the column became anaerobic during drainage, glucose was probably removed from the percolate by adsorption and anaerobic metabolism, as well as aerobically when oxygen was available.

6. A laboratory column dosed with settled sewage remained aerobic down to the capillary fringe. The first percolate after the capillary fringe was the most nitrified because it had been exposed to bacterial action for the longest time. The quality of the effluent then decreased until drainage rates dropped off sufficiently to allow more contact time between the system and substrate. Only slight nitrification occurred while the column was draining, although Kjeldahl nitrogen was diminished, probably by adsorption or filtration of nitrogenous substances.

7. Measurement of bacterial respiration rates and organic matter in laboratory columns showed that bacteria were most numerous near the surface. Their concentrations decreased rapidly with depth. Growth appeared to be limited mainly by a lack of oxygen.

#### 6-4 Practical Application

In designing an intermittent sand filter, an engineer seeks a high rate of infiltration and a high degree

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of treatment. He tries to attain these goals by proper design, which is achieved by choosing media having optimum characteristics, by determining the correct depth for the filter, and by selecting the desirable frequency and depth of wastewater application. These parameters also depend on the composition of the wastewater applied.

The results of this research cannot be applied directly to design a new facility; but they can be used to improve the operation of an existing installation or to aid in interpreting data from pilot-plant studies.

In most installations, observations of infiltration rates are used to determine the desired frequency and depth of application. No consideration is generally given to the quality of the percolate, if it is used for groundwater recharge, because of the difficulty in obtaining samples. Even when samples at various depths are collected in sampling pans, the results are questionable because the water so collected is not representative of the percolate in the soil. Clearly, the quality of the percolate is a major concern and should be determined when a groundwater basin is being recharged.

The maximum penetration of oxygen from the atmosphere into a typical spreading basin is only a few feet and it is almost constant for several days after a basin drains. A measure of this distance is valuable because it indicates the zone where nitrification and other aerobic activity

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occur. The distance can be measured by withdrawing gas samples from the soil atmosphere and analyzing them for oxygen. It can also be determined by taking a soil boring and analyzing the soil moisture for nitrate or by obtaining liquid samples with porous cups for nitrate analysis. Nitrate is high in the aerobic zone and decreases sharply where the soil becomes aerobic.

The maximum volume applied per cycle should be equal to the moisture retained in the aerobic zone after drainage to insure that all the wastewater will receive aerobic treatment. The frequency of application for complete nitrification can be determined by adjusting the frequency until analyses from core samples or from samples extracted through porous cups indicate that nitrification is essentially complete. Tests other than those for nitrate can be used whenever there is reason to believe that the concentration of some other contaminant is critical.

In pilot-plant studies and in installations where underdrains collect the percolate, gas determinations can be used to measure the oxygen profile. However, analyses of incremental samples of percolate also yield good information. Most studies on these systems have collected the entire percolate or a composite sample for chemical analysis. This procedure is poor because it yields little information suggesting how the system should be operated for better results. Incremental samples should be collected

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and analyzed to observe the variation of percolate quality with throughput.

Because oxygen is available only for the first few feet, samples collected from greater depths will not be high in oxidized products, such as nitrate, just after the surface is ponded. Percolate at greater depths will have passed through the upper few feet too quickly to become oxidized or while ponding caused the distance to become anaerobic. Nitrate and other oxidized products in the percolate at greater depths will increase as moisture retained at the top of the filter is displaced by the incoming liquid. Oxidized products will then decrease as newly added wastewater percolates to the bottom of the column or to the underdrain. A plot of the variation of oxidized products, such as nitrate, with throughput over several cycles will reveal a series of peaks. The volume added each cycle should be reduced until the volumes between the nitrate peaks become very small. The frequency of application should be adjusted to the maximum that will completely nitrify the volume retained.

The optimum media to be used in intermittent sand filters cannot be determined on the basis of this research. Some criteria for this media do present themselves, however. The media must be fine enough that its surface will be large and therefore support abundant microbial growth. On

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the other hand, it must be coarse enough to allow a high effective diffusivity for gases. Coarse media do not retain as much moisture as fine-textured media and maintain higher porosities when drained.

Depth to the water table or to underdrains should be enough that the capillary fringe is below the aerobic zone and does not decrease the effective porosity of that zone. <u>6-5 Suggestions for Further Studies</u>

1. This investigation has used nitrification as a major index of the efficiency of intermittent sand filters. Other components in reclaimed wastewaters may be more important, especially if they may be harmful to people drinking reclaimed water. Further research might well be conducted to determine the behavior of many more compounds than have been measured to date in wastewaters. Their removal and stabilization by percolation through soil systems should be determined.

2. Research is needed to allow engineers to design intermittent sand filters properly without the need for pilot-plant studies. Effective design also includes decisions relative to frequency and depth of application before the installation is built. Studies are necessary to determine the effects of variations in characteristics of porous media and wastewater composition on respiration rates. These data are needed to apply equation 2-13 to the design. Variation of substrate utilization with soil type also deserves consideration.

3. In one of the laboratory columns (No. 3), nitrification ceased when higher concentrations of glucose were added to the substrate. It is evident that heterotrophic bacteria outgrew the autotrophic nitrifying bacteria. When large quantities of carbonaceous substrate were added, it appears that carbonaceous bacteria were not forced to respire endogenously and consequently they multiplied heavily. Their generation times are so much less than those of the nitrifiers (30 minutes vs. 31 hours) that they completely enveloped the nitrifiers. These observations require further study.

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# NOTATION

	Dim	ensions are given in terms of mass (M), length (L),
and	time	(t). Symbols that appear infrequently or in one
section only are not listed.		
с	. =	total molar concentration, mols/L <sup>3</sup>
c <sub>A</sub>	=	molar concentration of species A, mols/L <sup>3</sup>
Ð	H	effective diffusivity, L <sup>2</sup> /t
D.	=	diffusivity in free space, L <sup>2</sup> /t
I	=	measured $\gamma$ -radiation with interference
Io	=	measured $\delta$ -radiation without interference
K	=	capillary conductivity, L/t
L	=	depth at which oxygen concentration becomes zero,L
NAz	=	molar flux of gas A in z direction with respect
		to stationary coordinates, mols/L <sup>2</sup> t
p	=	capillary head, L
q	=	respiratory quotient, dimensionless
RA	=	molar rate of production of gas A, mols/tL $^3$
t	=	time, t
×A	=	mole fraction of gas A, dimensionless
z	=	thickness of material, or soil depth, L
E	=	gas porosity, dimensionless
θ	=	soil water content, $L^3/L^3$
V	=	mass absorption coefficient of absorbing material for given energy of radiation, $L^2/M$
~	_	density of metorial W/r3
P	-	density of material, M/L
P'	=	bulk density of material, M/L <sup>3</sup>

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